10.3.2 User Newsletter

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This is the second user newsletter for Beamline 10.3.2. My apologies for not keeping everybody informed on a more frequent basis, but I've been busy. All this activity has allowed me to report some good news. The big development is that we've had our first user, Hoi-Ying Holman. She used 8 shifts to do XANES on Cr compounds and some unknowns. This marked the first time the beamline (in its present incarnation) was used to measure an unknown, and the first time it's done an EXAFS scan of something other than a pure metal foil. While no mapping was done, the micro capability was useful as the Cr in one of the unknowns was concentrated in small nodules. An unanticipated side benefit of a microbeam is that one doesn't have to be as careful as usual about thickness effects in EXAFS for concentrated powder samples. You simply pick the particle which happens to be the right thickness and put the beam there.

Now for a status update of the various systems which were being worked on at the time of the last newsletter:

Optics

The vertical collimating mirror, M2, was removed and rebent on the long-trace profilometer to an accuracy of better than 1μrad in slope. It was then put back and its tilt and translation adjusted. The beam divergence, as measured by scanning the second crystal pair against the first, comes out at 25 μrad, which is essentially the Darwin width for that monochromator. Thus, M2 seems to be working. However, an odd effect is that the best vertical spot size is obtained at a

slightly different M3 (vertical focus) tilt with white beam as opposed to monochromatic. Since the crystals are alike, it's not clear why that should be.

Some of our instabilities seem to be due to fluctuating temperature in the hutch. I've turned on the big overhead ventilation fan and that seems to have helped. The temperature now stays at 79-80^{o}F all the time, as long as the hutch door isn't opened too much. Crystal heating over a long period does seem to have an effect, so we've re-installed the cooling blocks after having removed the crystals for re-etching (*v.i.*).

The monochromator crystals were re-etched and tested for reflectivity. They check out with the theoretical Darwin width and >50% peak reflectivity. However, the transmission of the second crystal pair is still only 28% or so. The reasons for this are unclear, but anecdotal evidence suggests that this type of monochromator doesn't do better than that in practice.

The monochromator mechanics still need work. We can now do extended scans, which weren't possible before, but at the sacrifice of doing a backlash move on every step. This adds perhaps a second per point to the movement overhead, thus reducing the efficiency of data collection. We have also hung weights on various parts of the monochromator to make it cooperate.

During the April shutdown, we plan to do a complete rework of the mechanics of the mono. It will still be a 4-crystal mono, but it will use two Hubers rather than the tape-drive mechanism. The second Huber will be 'electronically geared' to the first. Electronic gearing is a system offered by National Instruments in which one motion axis is servoed to another so that when

one moves, the other moves by an amount equal to a precise multiple of the motion of the first axis. The ferrofluidic feedthroughs will also be replaced, probably by Teflon-sealed units. It may be possible to increase the bore size to 100mm, which would allow a Kryo-Tiger cryogenic system in place of the current water lines.

The maximum measured flux, with the full beam, was $3x10^9$ ph/sec at 7keV. This is still below the design specs, but suffices for many applications. During the April shutdown, it's possible we may get an extra 20-30% by moving the mask in front of the first mirror so that we get the whole beam for which the line was designed. At this point, the mask is perhaps a millimeter off.

Detector:

The 7-element detector is still out for repair, with no ETA as of yet. The software/firmware bug is still being worked on. We brought our setup to XIA and demonstrated the bugs to them. They then replaced piece after piece with their own stuff until the bugs were showing up with all their own equipment. This exercise exonerated the hardware. It now appears that the bugs are deep in the system design and can't be eradicated by simply changing the firmware. However, there are workarounds. For EXAFS, the symptom is that the detector will 'freeze' and keep reporting the same counts over and over. The fix for this is to re-initialize the detector every so often. The frequency with which this is required seems to depend on the count rate. Instead of re-initializing every 50 data points, another method is to do it when two successive counts come out

exactly equal. This approach is being tested right now. It does guarantee that one bad point will be taken when the detector freezes, but that's not too bad in EXAFS with multiple scans. With a fixed interval between resets, you can have large parts of the spectrum corrupted before the reset occurs. For mapping mode, the fix is to detect the occurrence of a problem and fix it by resetting and re-scanning the offending line. This fix has not yet been implemented.

Software:

The XY mapping is still 'almost there'. It does take data, but there are several bugs to be worked out. We hope to have it done by the time our second user comes in on November 2.

The EXAFS code and EXAFS data editor got a number of minor tweaks and bug fixes as a result of the run. Nothing major needed to be done.

Ease of use and miscellaneous issues

The optics chamber is now under vacuum ($2x10^{-6}$T). I have just installed a channeltron detector which looks at the Be exit window and picks up photoelectrons. This provides an I_0 monitor. This setup will be tested in two days when the beam comes back up from the Monday maintenance break. Before that, I was using a small Cu electrode in the air just below the exit window. This electrode and the window formed the plates of a tiny ion chamber, which I ran off a battery box. Unfortunately, this device isn't linear, so we're replacing it. This change should be transparent to the user as all the system cares about is that it sees a current source proportional to the incident intensity.

Full manuals have been written for the MCA utility and the EXAFS editor. Only a two-page ‘cheat sheet’ yet exists for the EXAFS data-taker.